



DRAFT INTERNATIONAL STANDARD ISO/DIS 15859-3

ISO/TC 20/SC 14

Secretariat: **ANSI**

Voting begins on
2002-09-12

Voting terminates on
2003-02-12

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Space systems — Fluid characteristics, sampling and test methods —

Part 3: Nitrogen

Systèmes spatiaux — Caractéristiques des fluides, échantillonnage et méthodes d'essai —

Partie 3: Azote

ICS 49.140

In accordance with the provisions of Council Resolution 15/1993 this document is circulated in the English language only.

Conformément aux dispositions de la Résolution du Conseil 15/1993, ce document est distribué en version anglaise seulement.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

Pour accélérer la distribution, le présent document est distribué tel qu'il est parvenu du secrétariat du comité. Le travail de rédaction et de composition de texte sera effectué au Secrétariat central de l'ISO au stade de publication.

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

Copyright notice

This ISO document is a Draft International Standard and is copyright-protected by ISO. Except as permitted under the applicable laws of the user's country, neither this ISO draft nor any extract from it may be reproduced, stored in a retrieval system or transmitted in any form or by any means, electronic, photocopying, recording or otherwise, without prior written permission being secured.

Requests for permission to reproduce should be addressed to either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Reproduction may be subject to royalty payments or a licensing agreement.

Violators may be prosecuted.

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Composition	2
4.1 Limits	2
4.2 Procurement	2
5 Fluid sampling	2
5.1 Plan	2
5.2 Responsibility for sampling	3
5.3 Sampling points	3
5.4 Sampling frequency	3
5.5 Sample size	3
5.6 Number of samples	5
5.7 Storage container	5
5.8 Gaseous samples	5
5.9 Liquid samples (vaporized)	5
5.10 Rejection	5
6 Test methods	5
6.1 General	5
6.2 Parameters of analysis	6
6.3 Nitrogen purity	6
6.4 Water content	6
6.5 Total hydrocarbon content (THC)	6
6.6 Oxygen content	7
6.7 Hydrogen content	7
6.8 Argon content	7
6.9 Carbon dioxide content	8
6.10 Carbon monoxide content	8
6.11 Aromatic, chlorinated, or halogenated hydrocarbon content	8
6.12 Nitrous oxide content	9
6.13 Odour	9
6.14 Particulate matter content	9
Annex A (informative) Gaseous chromatography (GC) and mass spectrometer (MS) applications	10

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 15859 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15859-3 was prepared by Technical Committee ISO/TC 20, *Aircraft and space vehicles*, Subcommittee SC 14, *Space systems and operations*.

ISO 15859 consists of the following parts, under the general title *Space systems — Fluid characteristics, sampling, and test methods*:

- *Part 1: Oxygen*
- *Part 2: Hydrogen*
- *Part 3: Nitrogen*
- *Part 4: Helium*
- *Part 5: Nitrogen tetroxide propellants*
- *Part 6: Monomethylhydrazine propellant*
- *Part 7: Hydrazine propellant*
- *Part 8: Kerosine propellant*
- *Part 9: Argon*
- *Part 10: Water*
- *Part 11: Ammonia*
- *Part 12: Carbon dioxide*
- *Part 13: Breathing air*

Introduction

This International Standard specifies limits for the composition of nitrogen and establishes the fluid sampling and test methods for nitrogen intended for use in the purging and pressurization of propellant systems of space systems. The purpose of this International Standard is to establish uniform requirements for the composition of nitrogen and the sampling and test methods for nitrogen used in the servicing of launch vehicles, spacecraft, and ground support equipment.

Fluid operations at a spaceport or launch site may involve a number of operators and supplier/customer interfaces, from the fluid production plant to the delivery to the launch vehicle or spacecraft. The fluid composition limits specified in this International Standard are intended to define the purity and impurity limits of the fluid for loading into the launch vehicle or spacecraft. The fluid sampling and test methods included in this International Standard are intended to be applied by any operator. The fluid sampling and test methods presented in this International Standard are acceptable methods for verification of the fluid composition limits.

Space systems — Fluid characteristics, sampling and test methods —

Part 3: Nitrogen

1 Scope

This part of ISO 15859 specifies limits for the composition of nitrogen and defines the fluid sampling and applicable test methods for verification of nitrogen composition. This International Standard establishes acceptable composition, test, and sampling requirements. This part of ISO 15859 applies to the following types and grades of nitrogen.

CAUTION — Gaseous nitrogen is an asphyxiant. Human contact with liquid nitrogen will result in severe injury. Care should be taken in the handling and storage of liquid nitrogen to prevent contact with the human body. Care should also be taken to prevent high concentrations of gaseous nitrogen in confined spaces.

- Type I: gaseous
 - grade A: purging/pressurizing;
 - grade B: crew breathing;
 - grade C;
 - grade F;
 - grade J;
- Type II: liquid
 - grade A: purging/pressurizing;
 - grade B: crew breathing;
 - grade C;
 - grade F.

This part of ISO 15859 is applicable to nitrogen used in both flight hardware and ground facilities, systems, and equipment. It is applicable to influents only to the extent specified herein.

This part of ISO 15859 is applicable to any sampling operation required to ensure that, when the fluid enters the launch vehicle or spacecraft, the fluid composition complies with the limits provided hereafter or with any technical specification agreed to for a particular use.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 15859. For dated references, subsequent amendments to, or revisions of, any of these publications

do not apply. However, parties to agreements based on this part of ISO 15859 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 8402:1994, *Quality management and quality assurance — Vocabulary*.

3 Terms and definitions

For the purposes of this part of ISO 15859, the terms and definitions given in ISO 8402 and the following apply.

3.1

total hydrocarbon content (as methane)

the single carbon atom equivalent

3.2

verification tests

analyses performed on the fluid in the container, or a sample thereof, which is representative of the supply

4 Composition

4.1 Limits

Unless otherwise provided in an applicable technical specification, the composition of nitrogen delivered to the flight vehicle interface shall be in accordance with the limits given in Table 1 when tested in accordance with the applicable test methods.

4.2 Procurement

Nitrogen types and grades specified in Clause 1 should be procured in accordance with an applicable national standard.

5 Fluid sampling

5.1 Plan

In order to ensure that the fluid composition complies with the limits specified in this International Standard, a fluid sampling plan should be established by all the involved operators, from the production to the space vehicle interface, and approved by the final user. Such plan shall specify:

- the sampling points;
- the sampling procedures;
- the sampling frequency;
- the sample size;
- the number of samples;
- the test methods;
- the responsibilities of any involved operator.

CAUTION — Gaseous nitrogen is an asphyxiant. Human contact with liquid nitrogen will result in severe injury. Care should be taken in the handling and storage of liquid nitrogen to prevent contact with the human body. Care should also be taken to prevent high concentrations of gaseous nitrogen in confined spaces.

5.2 Responsibility for sampling

Unless otherwise provided in an applicable technical specification, the nitrogen delivered to the flight vehicle interface shall be sampled and verified by the supplier responsible for providing the nitrogen to the flight vehicle. The supplier may use its own or any other resources suitable for the performance of the verification tests specified herein unless otherwise directed by the customer.

5.3 Sampling points

Unless otherwise specified, sampling shall be conducted at the fluid storage site or the flight vehicle interface.

5.4 Sampling frequency

Sampling shall be annually or in accordance with a time agreed upon by the supplier and the customer.

5.5 Sample size

The quantity in a single sample container shall be sufficient to perform the analysis for the limiting characteristics. If a single sample does not contain a sufficient quantity to perform all of the analyses for the required quality verification test, additional samples shall be taken under similar conditions.

Table 1 — Composition limits

Composition		Limits				
		Type I and type II (gaseous and liquid)				Type I
		Grade A	Grade B	Grade C	Grade F	Grade J
Nitrogen	volume fraction, a	99,5	f	99,995	99,9	99,999
Water	$\mu\text{L/L}$, max.	26,3	11,5	5,7	15	10
Total hydrocarbons as methane (CH_4)	$\mu\text{L/L}$, max.	58,3	5,0 ^g	5,0	25	—
Oxygen	$\mu\text{L/L}$, max.	5 000	50	20	1 000	5
Hydrogen	$\mu\text{L/L}$	e	e	0,5	e	—
Argon ^b	$\mu\text{L/L}$	e	20	2,0	e	—
Carbon dioxide ^b	$\mu\text{L/L}$, max.	e	5,0	5,0	e	—
Carbon monoxide ^b	$\mu\text{L/L}$, max.	e	5,0	5,0	e	—
Aromatic hydrocarbons as benzene ^b	$\mu\text{L/L}$, max.	—	0,5	—	—	—
Halogenated hydrocarbons ^b	$\mu\text{L/L}$, max.	—	1,0	—	—	—
Chlorinated hydrocarbons ^b	$\mu\text{L/L}$, max.	—	0,1	—	—	—
Nitrous oxide ^b	$\mu\text{L/L}$, max.	—	1,0	—	—	—
Odour		—	None	—	—	—
Particulate matter ^c	mg/L, max.	—	—	1,0	1,0	—
Total impurities ^d	$\mu\text{L/L}$, max.	5 000	100	50	1 000	10
<p>^a Percent nitrogen includes trace quantities of neon, helium, and small amounts of argon.</p> <p>^b If required by use.</p> <p>^c Applies only to Type II (liquid) nitrogen.</p> <p>^d Sum of all percentages of water, hydrocarbons, oxygen, hydrogen, and if applicable, carbon monoxide, carbon dioxide, and argon.</p> <p>^e No limit for this grade.</p> <p>^f By indirect method 99,99 or by direct method 95,0.</p> <p>^g For Environmental Control and Life Support System (ECLSS) ground test only.</p>						

5.6 Number of samples

The number of samples shall be in accordance with one of the following:

- a) one sample per storage container;
- b) any number of samples agreed upon by the supplier and the customer.

5.7 Storage container

Unless otherwise provided by the applicable sampling plan, the fluid storage container shall not be refilled after the time the sample is taken.

5.8 Gaseous samples

Gaseous samples shall be a typical specimen from the gaseous nitrogen supply. For safety reasons, the sample container and sampling system must have a rated service pressure at least equal to the pressure in the supply container. Samples shall be obtained in accordance with one of the following:

- a) by filling the sample container and storage containers at the same time, on the same manifold, and under the same conditions and with the same procedure;
- b) by withdrawing a sample from the supply container through a suitable connection into the sample container. No pressure regulator shall be used between the supply and the sample containers (suitable valves are permissible);
- c) By connecting the container being sampled directly to the analytical equipment using suitable pressure regulation to prevent over-pressurizing this equipment.

5.9 Liquid samples (vaporized)

Vaporized liquid samples shall be a typical specimen from the liquid nitrogen supply. Samples shall be obtained by flowing liquid from the supply container into or through a suitable container in which a representative liquid sample is collected and then completely vaporized.

5.10 Rejection

When any sample of the fluid tested in accordance with Clause 6 of this International Standard fails to conform to the requirements specified herein, the fluid represented by the sample shall be rejected. Disposition of the rejected fluid shall be specified by the customer.

6 Test methods

6.1 General

The supplier will ensure, by standard practice, the quality level of nitrogen. If required, alternate test methods are described in Clause 6 of this International Standard. Other test methods not listed in this International Standard are acceptable if agreed upon between the supplier and the customer.

These tests are a single analysis or a series of analyses performed on the fluid to ensure the reliability of the storage facility to supply the required quality level. This can be verified by analysis of representative samples of the fluid from the facility at appropriate intervals as agreed upon between supplier and the customer. Tests may be performed by the supplier or by a laboratory agreed upon between the supplier and the customer.

The analytical requirements for the tests shall include the determination of all limiting characteristics of nitrogen.

6.2 Parameters of analysis

The parameters for analytical techniques contained in this section are:

- a) purity contents shall be expressed as a percentage (%) by volume unless otherwise specified;
- b) calibration gas standards containing the applicable gaseous components may be required to calibrate the analytical instruments used to determine the limiting characteristic levels of the fluid;
- c) if required by the customer, the accuracy of the measuring equipment used in preparing these standards shall be traceable to an established institute for standards;
- d) analytical equipment shall be operated in accordance with the manufacturer's instructions;
- e) analytical methods not listed in this International Standard are acceptable if agreed upon between the supplier and the customer.

6.3 Nitrogen purity

The nitrogen concentration shall be determined by one of the following procedures:

- a) by determining the amount of the aggregate impurities using the methods in the following sections. The percent nitrogen is the value obtained when this amount, expressed as percent, is subtracted from 100;
- b) by determining the aggregate of all impurities by mass spectrometer. The percent nitrogen is the value obtained when the aggregate, expressed as percent, is subtracted from 100.
- c) by a gas chromatographic method which uses a 5A zeolite column and measures the peak height versus retention time;
- d) by a mass spectrometer method which measures nitrogen at an atomic mass unit (amu) of 28. Carbon monoxide also has an amu of 28 but it can be determined by the method described under 6.10.

6.4 Water content

For liquid nitrogen, the water content cannot be determined by sampling, only online measurements are possible. The water content shall be determined by one of the following procedures:

- a) by a dewpoint analyzer in which the temperature of a viewed surface is measured at the time water first begins to form;
- b) by a piezoelectric sorption hygrometer, of which the accuracy of analysis shall be plus or minus 0,1 cubic centimetre per cubic metre or 5 % of the reading, whichever is greater;
- c) by a metal oxide capacitor equipped analyzer on a range which is no greater than 10 times the specific maximum water content;
- d) by an electrolytic hygrometer having an indicator graduated in cubic centimetres per cubic metre on a range which is not greater than 10 times the specified maximum water content.

6.5 Total hydrocarbon content (THC)

The total (volatile) hydrocarbon content (as methane) shall be determined by one of the following procedures:

- a) by a flame ionization-type analyzer. The analyzer shall be calibrated at appropriate intervals by the use of calibration gas standards. The range used shall be no greater than 10 times the specified maximum total hydrocarbon content expressed as methane;

- b) by a gas cell-equipped infrared analyzer. The analyzer shall be calibrated at appropriate intervals by use of calibration gas standards at a wavelength of approximately 3,5 micrometre (the characteristic absorption wavelength for C-H stretching). The analyzer shall be operated so that its sensitivity for methane is at least 10 % of the specified maximum total hydrocarbon contents;
- c) by a gas chromatograph in accordance with 6.8 of this International Standard.

6.6 Oxygen content

The oxygen content shall be determined by one of the following procedures:

- a) by an electrochemical-type oxygen analyzer containing a solid or an aqueous electrolyte. The analyzer shall be calibrated at appropriate intervals by use of calibration gas standards or integrally in accordance with Faraday's Law. The range used should be no greater than 10 times the specified maximum oxygen content;
- b) by a tungsten filament lamp-type analyzer. The analyzer shall be checked for proper operation at appropriate intervals by the use of a calibration gas standard;
- c) by an analyzer in which oxygen reacts to form a compound which is subsequently measured. The analyzer shall be calibrated at appropriate intervals by the use of calibration standards. The range used shall be no greater than 10 times the specified maximum oxygen content;
- d) by a gas chromatograph method such as that described under 6.8 of this International Standard;
- e) by a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified oxygen content;
- f) by an apparatus employing a detector tube filled with a color-reactive chemical. The degree of accuracy is dependent on the precision of the measurements and the analytical bias of the tube;
- g) by a heated zirconium oxide-type analyzer or by a paramagnetic-type analyzer. The analyzer shall be calibrated at appropriate intervals by the use of calibration gas standards. The range used shall be no greater than 10 times the specified maximum oxygen content.

6.7 Hydrogen content

The hydrogen content shall be determined by one of the following procedures:

- a) by a gas chromatograph in accordance with 6.8 of this International Standard;
- b) by a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component;
- c) by an analyzer in which hydrogen reacts to form a compound which is subsequently measured. The analyzer shall be calibrated at appropriate intervals by the use of calibration gas standards. The range used shall be no greater than 10 times the specified maximum hydrogen content.

6.8 Argon content

The argon content shall be determined by one of the following procedures:

- a) by a gas chromatograph. This method may be used not only for argon, helium, and neon determination but also for the determination of any other limiting characteristic gaseous components. The analyzer shall be capable of separating and detecting the component with a sensitivity of 10 % of the specified maximum amount of the component. Appropriate impurity concentrating techniques may be used to attain the sensitivity. The analyzer shall be calibrated at appropriate intervals by the use of calibration gas standards;

- b) by a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component.

6.9 Carbon dioxide content

The carbon dioxide content shall be determined by one of the following procedures:

- a) by a gas cell-equipped infrared analyzer. The analyzer shall be calibrated at appropriate intervals by the use of calibration gas standards at a wavelength of approximately 4,3 micrometre. The analyzer shall be operated so its sensitivity for carbon dioxide is at least 10 % of the specified maximum carbon dioxide content;
- b) by a gas chromatograph method such as that described in 6.8 of this International Standard. The technique utilized shall be specific for the separation and analysis of carbon dioxide;
- c) by an apparatus employing a detector tube filled with a color-reactive chemical. The degree of accuracy is dependent on the precision of the measurements and the analytical bias of the tube;
- d) by a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component;
- e) by a catalytic methanator gas chromatograph method such as that described under 6.8 of this International Standard.

6.10 Carbon monoxide content

The carbon monoxide content shall be determined by one of the following procedures:

- a) by an apparatus employing a detector tube filled with a color-reactive chemical. The degree of accuracy is dependent on the precision of the measurements and analytical bias of the tube;
- b) by a gas cell-equipped infrared analyzer. The analyzer shall be calibrated at appropriate intervals by the use of calibration gas standards at approximately 4,6 micrometre. The analyzer shall be operated so that its sensitivity for carbon monoxide is at least 10 % of the specified maximum carbon monoxide content;
- c) by an analyzer in which carbon monoxide reacts to form a compound which is subsequently measured. The analyzer shall be calibrated at appropriate intervals by the use of calibration standards. The range used shall be no greater than 10 times the specified maximum carbon monoxide content;
- d) by a gas chromatograph method such as that described under 6.8 of this International Standard. The technique utilized shall be specific for separation and analysis of carbon monoxide;
- e) by a catalytic methanator gas chromatograph method such as that described under 6.8 of this International Standard.

6.11 Aromatic, chlorinated, or halogenated hydrocarbon content

The aromatic, chlorinated, or halogenated hydrocarbon content shall be determined by one of the following procedures:

- a) by a gas chromatograph method such as described under 6.8 of this International Standard;
- b) by a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component;
- c) by a gas chromatograph/mass spectroscopy analyser. The analyser shall be calibrated at appropriate intervals by use of calibration gas standards. The analyser shall be operated so that its sensitivity for methane is at least 10 % of the specified maximum hydrocarbon contents.

6.12 Nitrous oxide content

The nitrous oxide content shall be determined by one of the following procedures:

- a) by a gas cell-equipped infrared analyzer. The analyzer shall be calibrated at appropriate intervals by the use of calibration standards at approximately 4,5 micrometre (the characteristic absorption wavelength for nitrous oxide). The analyzer shall be operated so that its sensitivity for nitrous oxide is at least the specified maximum amount;
- b) by a gas chromatograph in accordance with 6.8 of this International Standard.

6.13 Odour

The odour shall be determined by the following procedure:

- a) odour in liquid is checked by evaporating to dryness 200 mL of liquid in a loosely covered 400-mL beaker or similar container with a fresh filter paper in the bottom. The cover is removed at the point of complete evaporation, and the beaker is odour tested several times until it has warmed to above the freezing point of condensed water on the outside.

CAUTION — In the above procedure, do not place face directly in front of the valve or beaker. Instead, cup the hand and bring some of the gas being vented toward the nose.

6.14 Particulate matter content

A particulate matter test method is not required for gaseous nitrogen. The particulate matter of liquid nitrogen shall be determined by the following method: pass a liquid nitrogen sample through a specified low micrometre-rated, tare-weighted, analytical filter disk contained in a suitable holder. The filtered liquid is collected and measured in an open container. The assembly is warmed and the disk is reweighed. The size of any particulate matter can be evaluated by the examination of the filter disk with a suitable optical magnifier.

Annex A (informative)

Gaseous chromatography (GC) and mass spectrometer (MS) applications

Gaseous chromatography (GC) should be used as the reference or preferred method to analyze nitrogen impurities, except for odour, water, and particulate content.

A mass spectrometer plus gaseous chromatography (MS + GC) may be used as an alternative to gaseous phase chromatography to avoid possible interference (especially for the hydrocarbons).

Table A.1 summarizes the applications of these methods for nitrogen. "X" indicates that the method is to be used and "—" indicates that it is not used.

Table A.1 — Application of GC and MS

Characteristic	GC with DID detector on molecular sieve column	GC with FID detector on porapack column (or equivalent)	GC with methanator + FID on porapack column (or equivalent)	MS + GC	MS
	—	—	—	—	—
Total hydrocarbon content	—	X	—	X	—
Oxygen	—	—	X	X	—
Hydrogen	—	—	X	X	—
Argon	X	—	—	X	X
Carbon dioxide	—	—	X	X	—
Carbon monoxide	X	—	X	X	—
Aromatic, chlorinated, halogenated hydrocarbons	—	X ^a	—	X	—
Nitrous oxide	X	—	—	X	—
Odour	—	—	—	—	—
Particulate matter	—	—	—	—	—
^a An electron capture detector (ECD) could also be used for halogenated hydrocarbons detection.					